AMSTE-TM-T (70) 13 Aug 96

MEMORANDUM FOR Administrator, Defense Technical Information Center, ATTN: 8725 John J. Kingman Rd., STE 0944, Ft Belvoir, VA 22060-6218

SUBJECT: Test Operations Procedure (TOP) 2-2-690, Army Oil Analysis Program for Vehicle Testing, 16 Aug 96

1. Enclosed are DTIC Form 50 (Encl 1) and two copies of subject test operations procedure (Encl 2) for assignment of accession number.

2. The TECOM point of contact is Mr. Wolfgang HR. Schmidt, AMSTE-TM-T, amstectt@apg-9.apg.army.mil, DSN 298-1486.

FOR THE COMMANDER:

2 Encls

C. DAVID BROWN, Ph.D.
Chief, Simulation & Technology Div
Directorate for Technical Mission

Encl 2

DEPARTMENT OF THE ARMY
HEADQUARTERS, U.S. ARMY TEST AND EVALUATION COMMAND
ABERDEEN PROVING GROUND, MARYLAND 21005-5055

REPLY TO
ATTENTION OF

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13. ABSTRACT *(Maximum 200 words)*
This TOP describes the procedures for oil program for vehicle testing.

14. SUBJECT TERMS
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Acid/Base Number
Particle Count
Spectroscopy
Ferrography
Titrator

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U.S. ARMY TEST AND EVALUATION COMMAND
TEST OPERATIONS PROCEDURE

Test Operations Procedure (TOP) 2-2-690 16 August 1996
AD No.

ARMY OIL ANALYSIS PROGRAM FOR VEHICLE TESTING

1. SCOPE. This TOP describes the procedures for conducting oil analysis during vehicle testing to include the following:

a. Physical Property Tests: water contamination, viscosity, acid/base number, fuel contamination by Fourier Transform Infrared (FTIR) Analysis.

b. Particle Count Analysis: on hydraulic fluid used in hydraulic systems.

c. Spectrometric Oil Analysis: wear metals in lubricating oil, additive packages depletion, identification of other non-metal contaminants such as silicon and sodium.

d. Ferrography: identification of wear metal particles for characterization (type of wear), foreign contamination (grease, coal, asbestos, carbon flakes), and quantitative analysis (wear particle concentration of large size particles to small size) to evaluate accelerated wear preceding component failure.

e. Fourier Transform Infrared Analysis: identification of oil type, monitoring additive package depletion, contamination by water, fuel, antifreeze, monitoring soot buildup and oil degradation.

Oil analysis information provides interval monitoring of the quality of the oil taken from various fluid-wetted components. Results will aid in the early detection of impending failures. Overall oil analysis information collected can be incorporated into the Army Oil Analysis Program (AOAP) groundwork for establishing routine testing criteria and sampling intervals

for a specific vehicle or vehicle type.

2. FACILITIES AND INSTRUMENTATION.

2.1 Facilities.

Item	Requirement
Petroleum, oils and lubricants (POL) laboratory	Dust-free environment (environmentally controlled preferred)
Fume hoods	Flow rate 2.8 m^3/min (100 ft^3/min)
Waste handling operation	Designated hazardous waste satellite site
Solvents	Chloroflourocarbon (CFC) free solvents (i.e., ELECTRON, ACTISol solvent blend)
Oven	60 °C range
Analytical laboratory	Environmental control 24 °C ± 2 °C <50% humidity
Sample storage shed	Appropriate to hold sample load
Others as required by the test plan	

2.2 Instrumentation.

Devices for Measuring	Measurement Accuracy
Physical test properties of oils:	
Viscosity (viscometer)	±0.3 centistokes
Percent H_2O	±0.001%
Acid/base number (titrator)	±0.2%
Particulate contaminants	Size range 5 - 100 μm
Spectrometric analysis of oils:	

Wear metals (spectrometer)	95% confidence limits
Wear particle assessment in oils:	
Wear identification direct reader (ferrography)	Required accuracy is dependent upon specific test requirements and should be addressed at that time
Identification of contaminants in oils:	
FTIR spectroscopy	1 cm^{-1} resolution 4000 to 400 cm^{-1}

3. REQUIRED TEST CONDITIONS.

3.1 Field Test Planning.

a. Inform laboratory personnel of incoming vehicle test items requiring oil analysis. This is to be done through a memorandum with background information (type of oil to be used throughout seasonal testing) and any lube orders that might accompany the vehicles.

b. Ensure that upon arrival of the vehicle, during the initial servicing, an oil sample is taken and submitted to the laboratory for baseline or identification checks. Give particular attention to the engine and transmission systems. During initial or routine inspection, a sample from the original container used to replenish the lubricants should be submitted. Many manufacturers use various kinds/concentrations of additive packages, making it difficult to establish a good trend. Therefore, it is recommended that a virgin oil sample be submitted to ensure that the correct oil is used.

c. Establish sampling points according to the lube orders and follow sampling technique as outlined in TB-43-0211[1*] and TM 38-301-2[2].

d. Prelabel oil bottles (NSN 8125-01-082-9697) with pertinent information (i.e., vehicle identification, model number, date collected, engine hours/miles since last oil change, amount of oil added since last sample, and test director). Include on the label whether the sample is collected cold or hot. Hydraulic fluid samples requiring particle contamination analysis must be collected in clean, particle-free sample bottles. Sample bottles that have not been cleaned to specifications may contain residual particles which will give false readings. Clean sample bottles can be obtained from the laboratory.

*Superscript numbers correspond to those in Appendix D, References.

e. Obtain samples from all lubricant/fluid reservoirs as indicated by the lubrication order for the vehicle. Samples should be taken within a short period after the component or system has been operating to ensure that a representative sample of circulated oil is taken.

f. Submit all oil samples to the laboratory within a 24-hour time frame. This will ensure sample integrity.

g. Provide the laboratory with pertinent information acquired during servicing/maintenance. This information will be used to assist the laboratory in making decisions relative to when changeout is needed.

3.2 Laboratory Test Plan.

a. Log oil samples into sample log data base along with pertinent information (see para 3.1d). Assign a laboratory number to each sample submitted. Inspect sample bottles and verify that the labels are filled out correctly and the bottle lids are secured.

b. Visually inspect the oil samples for water or coolant contamination and record accordingly. The free water in the oil will settle to the bottom of the bottle. Oil with excess water will appear grayish in color. Record any unusual appearances of the samples.

c. Preheat laboratory oven to a temperature of 60 °C ± 5 °C. Once the oven has reached the desired temperature, place samples into the oven and allow to heat for a maximum of 30 minutes.

d. Allow any spectrometers, viscometers, and titrators a 1-hour warm up period prior to testings.

e. Prepare all standards and reagents to be used in testing.

f. Verify that the viscometer has reached the required temperature as outlined in the test procedure.

g. Obtain laboratory data sheets to be used for each test procedure.

h. Verify criteria, for viscosity and acid/base number, which will be used to rate individual oil samples. This information can be found in military specifications.

4. TEST PROCEDURE.

a. Physical Properties: Analyze all oil samples for water and viscosity. Only the oil samples from the engine and transmission will require acid/base number titrations. Particle count analyses are performed on hydraulic fluids from reservoirs and hydraulic systems. Conduct these tests in the following manner:

(1) Water determination: Karl Fischer method (TM 38-301-2 or ASTM-D1744[3]): Set up the Karl Fischer titrator in accordance with the manufacturer's operating procedures[4]. Check the balance before starting, clear the balance if necessary, and wipe the pan clean. Shake sample vigorously. Using a pasteur pipet, measure out approximately 2 ml of oil sample. Wipe the pipet clean of any excess oil. Zero the balance before placing the pipet on the balance. After placing the pipet with the sample on the balance, tare the balance. The titrator will automatically titrate out residual water in the solvent. Dispense a few drops into the vessel with the solvent. Follow manufacturer's directions for operation of the instrument. (Respond to the prompts on the display of the titrator.) Results are recorded in percent. Record this data on the data sheet.

Note: This method is only to be used as a semiquantitative measurement for MIL-L-2104 lube oil. For lower detection limit, the FTIR should be implemented.

(2) Kinematic viscosity (ASTM-D445[5]): Heat oil samples in a dry oven set at 60 °C ± 5 °C for 30 minutes. Stir and shake sample until waxy material is mixed into the sample. Dispense 20 ml of oil sample into a disposable cup used on the automatic viscometer. Follow the manufacturer's directions for specific instrument operation in setting up the automatic viscometer. Load automatic carousel with oil samples. Once the viscometer is brought to operating temperature, the samples will be analyzed automatically. Each sample will be measured several times until readings are obtained that are within the tolerance range set for the instrument. An established value is ±0.35% of one another. Once the program has ceased, record all data in centistokes on the data sheets.

(3) Acid/base number (ASTM-D664[6]): Set up the titrator in accordance with the manufacturer's instructions. Tare the balance before starting. Heat the oil sample to 60 °C ± 5 °C until all of the sediment is homogeneously suspended in the oil. Place an empty disposable titration beaker on the balance and tare the balance. Shake the sample vigorously. Weigh out 5.0 ± 0.5 grams of oil into the titration beaker. Enter the weight into the titrator. The weight may transfer automatically if using an automated titrator. Place the beaker with sample on the stand or carousel. Make sure the electrode is half immersed. Start the stirrer and titrator using 0.1 N alcoholic NaOH (sodium hydroxide) solution. Once the acid/base number is obtained, the results will be printed out. Record this value on the sample data sheet.

(4) Particle count analysis (MIL-H-6083[7], MIL-H-83282[8], MIL-H-46170[9]): All samples are collected into clean, particle-free glass bottles. Set up instrument in accordance with manufacturer's specifications as prescribed for a particular fluid. Flush system with mineral spirits before using. Shake each sample before placing into the sample chamber. After the sample is secured in the chamber, apply vacuum to the system for approximately 2 minutes. This will aid in eliminating air bubbles. Once the

bubbles are removed, turn off the vacuum. Set instrument to apply pressure to the system. The pressure should be 207 kPa (30 psi) with a suction rate of 10 ml per 7 seconds. Set parameters as follows:

Four runs
Average
Counts/100 ml
Cumulative versus differential
Channels desired

Once four readings have been obtained and averaged, record the average on the data sheet. Evaluate data by comparing with military standards for the oil of interest.

Verify that all data are accurate and reasonable. (Quality control checks should be made periodically for accuracy and repeatability.) Compare results with military specifications for viscosity and acid/base number when stated.

b. Spectroscopy (TM 38-301-2): Spark Emission: Heat oil samples in oven at 60 °C ± 10 °C or place them on a hot plate set at 49 °C for 10 minutes. Shake containers vigorously until all sediment is homogeneously suspended in the oil. Turn the fume hood on. Set up the instrument in accordance with manufacturer's manual[10]. Allow 45 minutes for warmup. Follow manufacturer's operational procedure for calibrations. Pour out five standards of any concentration into oil caps (Federal stock number (NSN 6640-01-042-6583)). Burn each standard and note the concentration of each burn. If the concentrations are within the limits of the instrument, proceed with samples. If the standards fall outside calibration limits for the instrument, recalibrate the instrument following operating procedures. Once the instrument has been verified as being calibrated, begin sample analysis. Pour each sample into a clean cap. Before each burn, the electrode rod and disc must be replaced. After inserting new rods, verify the analytical gap between rod and disc. If the instrument is in automatic mode, results will transfer automatically into a PC data base. If the instrument is set in manual mode, record data for each element by rotating the knob located on the front panel. Perform quality control checks every ten sample analyses by checking the calibration standard for accuracy (±0.5%).

c. Ferrography (Wear Particle Analysis): Heat oil in oven at 60 °C ± 10 °C or place on hot plate set at 49 °C for 10 minutes. Shake container vigorously until all sediment is homogeneously suspended in the oil. Assemble precipitator tube into position on the direct reader (see operational manual[11] for setting up). Dispense 1 ml of sample and 3 ml of fixer solution into a glass test tube. Shake the sample vigorously for homogeneity. The sample should be analyzed within minutes of mixing; otherwise, the heavy particles will precipitate out and could affect the reading. Entry end is placed in the test tube and the glass portion of the tube is clamped under a magnet. Push "start" to initiate the flow of the oil through the precipitator

tube. Once the complete sample has passed through the tube, note the reading on the display. Record the number displayed for large and small particles. If there is a dramatic increase in the direct reading, the sample should be analyzed qualitatively. Prepare a slide by measuring out 2 ml of oil into a test tube. Dilute the sample with 2 ml of fixer to improve precipitation and adhesion. Carefully open a substrate (glass slide) without touching the substrate. Insert the slide on the magnetic platform. The lip of the slide should touch the drain tube. Press "start" to begin the flow of the sample. The sample will flow over a magnetic field. Once the flow subsides, the substrate is washed with a solvent to remove residual oil, leaving behind embedded particles. Allow the slide to dry before removing. Lift the slide straight up and place into its protective holder. The slide is then observed under a bichromatic microscope for evaluation comparison to the defined text in Table 1.

d. Fourier Transform Infrared Spectroscopy: Set up the spectrometer following the manufacturer's procedure[12] for alignment and calibration check. If a sample is two layers, the layers should be analyzed separately. Shake sample vigorously to ensure a homogeneous mixture. Pour oil onto the surface of a horizontal attenuated total reflectance (ATR) crystal. The oil analysis is started using batch files (macros) to subtract out the background, integrate and calculate concentration of antioxidant, hydroxyl, soot, oxidation and fuel levels found in the oil sample.

TABLE 1. SUGGESTED PROCEDURE FOR ANALYSIS OF A FERROGRAM

Step	Mag.[a]	Ref.[b]	Trans.[c]	Comments
1	100x	Red Filter	Green Filter	Look for severe wear particles at entry by presence of bright red. Normal rubbing wear particles are too small to be resolved at this magnification and will appear black. Therefore, if only normal rubbing wear particles are present on the ferrogram, no red particles will be observed. Scan length of ferrogram looking for severe nonferrous wear particles, nonmetallic particles, or a heavy deposit at the exit typical of corrosive wear.
2	500x	White Light	Green Filter	Examine the entry deposit making a preliminary judgment as to the specific types of wear particles present such as severe wear, normal rubbing wear, chunks, etc. A preliminary judgment of dark metallo-oxides must be confirmed at 1000x magnification because particles that are not flat will appear dark. Scan the length of the ferrogram looking for nonferrous metal particles and other distinctive features such as nonmetallic

Step	Mag.[a]	Ref.[b]	Trans.[c]	Comments
				particles, friction polymers, etc.
3	800x	White Light	Green Filter	Most particle types can be recognized at 400x, but 1000x provides critical details necessary to complete the analysis. Spheres, fine cutting wear particles, and small spots of temper color on the surface of particles indicative of high heat during generation can be distinguished only at high magnification. Because of the high numerical aperture of the 100x lens (good light-gathering ability), jagged free-metal particles may be distinguished from dark metallo-oxides. Nonmetallic amorphous particles do not contain fine metal particles whereas friction polymers are recognized by the presence of fine metal particles in the amorphous matrix. Bichromatic light (red reflected and green transmitted) is useful for identifying friction polymers because of the greater contrast provided. When using the 100x objective, it is recommended that one hand be used for the fine focus control so that the stage may be easily racked up or down. When viewing particles that are not perfectly flat, continually move the stage so that focus scans up and down, forming a more complete impression of the particles.
4	100x	OFF	POL	Use polarized transmitted light to identify nonmetallic particles. These will appear bright in an otherwise dark field.
5	500x	POL	OFF	Use polarized reflected light to determine surface characteristics of particles. Oxidized surfaces of metal particles will depolarize the light. Small nonmetallic particles, which may not have been seen at 100% magnification can now be detected. Use 1000x magnification with polarized reflected light if surface characteristics are of particular interest.
6	As Required			Take photos prior to heat treating ferrogram. A polarized light photo may be appropriate if it is desired to distinguish between organic and inorganic birefringent particles. The organic particles will not be as bright after

Step	Mag.[a]	Ref.[b]	Trans.[c]	Comments
				heat treatment. A second photo exposed for the same time will show this difference. It may also be desirable to photograph strings of ferrous particles prior to heat treatment as well as any suspected Pb/Sn alloy particles.
7		-		Heat treat ferrogram.
8		As Required		Reexamine ferrogram after heat treating. Take photos as necessary.
9		-		Heat treat to higher temperature if required.

[a]Magnification.
[b]Reflected.
[c]Transmitted.

5. DATA REQUIRED.

a. Water: Karl Fischer percent water-- (% H_2O).

b. Kinematic viscosity --centistokes (cSt).

c. Acid/base number relative units-- total acid number/total base number (TAN/TBN).

d. Particle count analysis (cumulative versus differential)-- particles per 100 milliliters.

e. Wear metals and additive depletion analysis --parts per million (PPM) Fe, Ag, Al, Cr, Cu, Mg, Na, Ni, Pb, Si, Sn, Ti, Zn and B.

f. Ferrography -- Direct reading of large versus small particles, pictures of particles appearing at the entrance, middle and exit end of the ferrogram. Record observations at various magnifications of microscope.

g. Fourier Transform Infrared Spectrometer-- Submit spectrum of samples with labeled contamination peaks, such as soot, water, glycol and grease.

6. PRESENTATION OF DATA.

a. Present all data in a narrative, tabular or chart format as appropriate. Data for wear metals and additive depletion, water, viscosity, acid/base number are recorded on data sheets listed in Appendix A.

b. Present spectrums and photographs for samples showing specific problems and contaminants (see app B and C).

c. Review quality control data when appropriate to verify the integrity of the analysis performed.

d. All data for ongoing projects should be placed into a data base for easier review of trend analysis.

e. A printout of all information obtained from the analysis: wear metals, water, sediment, and viscosity will be given to the test director upon completion of the analysis. (The laboratory has set a three-day turn-around time for POL analysis.)

APPENDIX A. SAMPLE DATA SHEETS

TEST DATA
(SOP MP 70-6)

DATE:_____
SHEET NO._____

Sample Number		% Water		% Sediment		cSt Viscosity			TAN/ TBN

Figure A-1. Physical property test data sheet.

Sample Number	FE	AG	AL	CR	CU	MG	NA	NI	PB	SI	SN	TI	B	MO	ZN

Figure A-2. Wear metal analysis data sheet.

APPENDIX B. EXCERPT OF REGION OF INTEREST FOR FTIR SPECTRUM

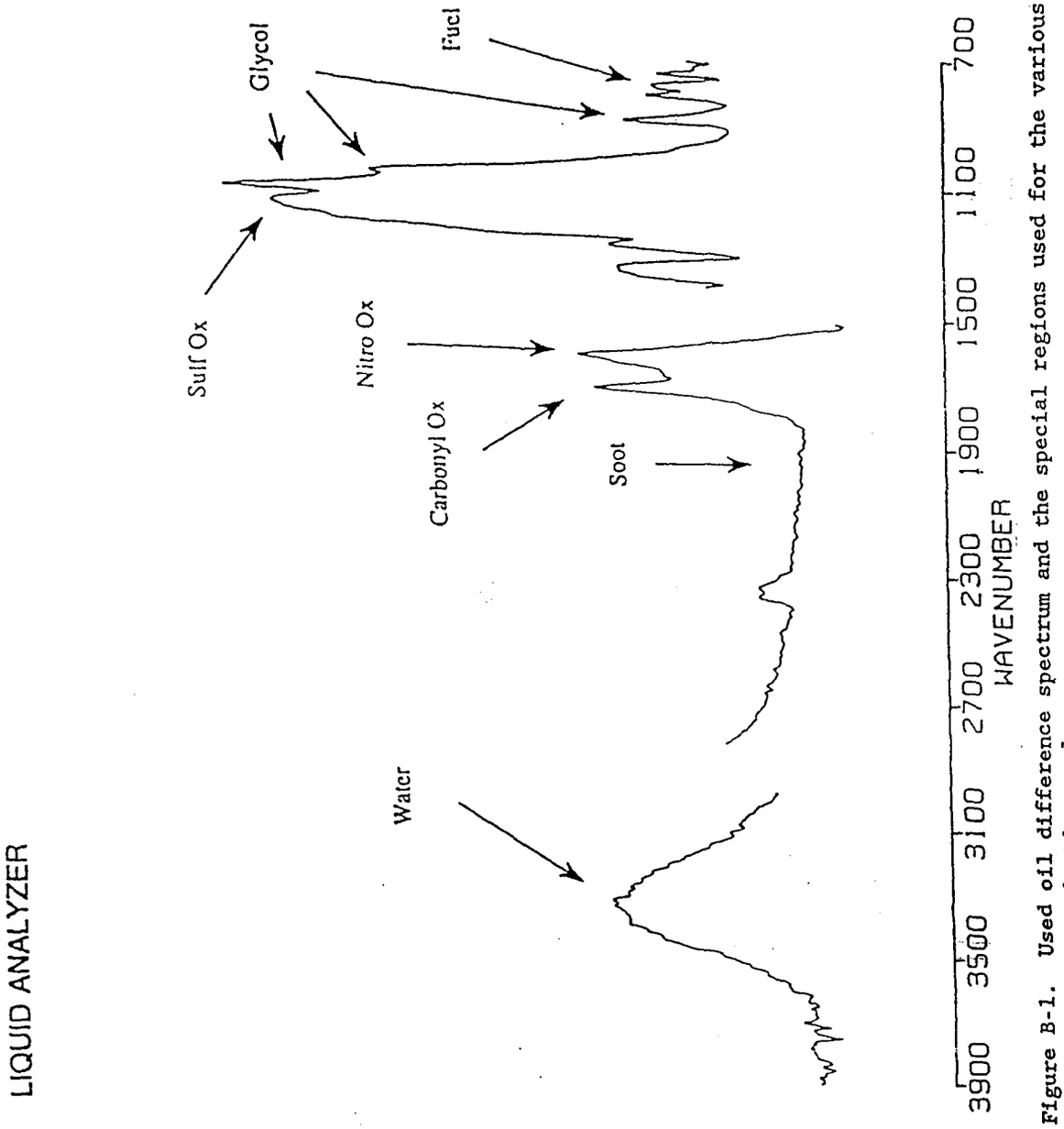
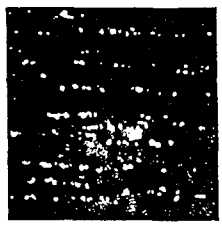
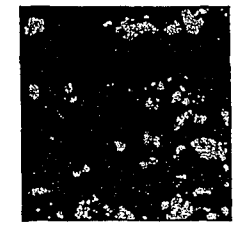
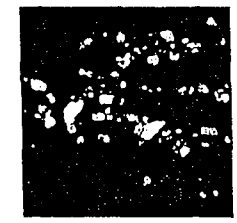


Figure B-1. Used oil difference spectrum and the special regions used for the various quantitative analyses.

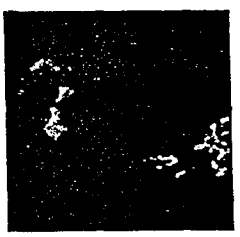
APPENDIX C. EXAMPLE OF PHOTOS FROM FERROGRAPHY

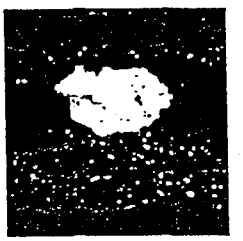
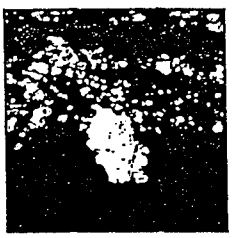
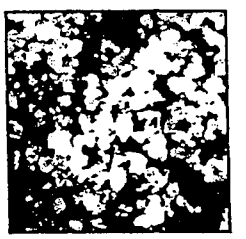
TYPICAL MICROSCOPIC WEAR PARTICLES OBSERVED

Actual Photos	Description	Examples of Causes
 (1). Normal Rubbing Wear	Flat platlets < 5 µm in major dimension. Magnification 200x	Normal machine wear.
 (2). Severe Sliding Wear	Flat particles > 20 µm with striation. Magnification 1000x	Excessive load/speed on sliding surface.
	Long, curled strips of metal. Magnification 1000x	Misalignment or abrasive contamination in lubricant.
 7). Molybdenum Disulfide	Nonferrous particle, gray in color with many shear planes. Magnification 400x	Solid lubricant additive in system.

Actual Photos	Description	Examples of Causes
 (7). Black Oxides	Black particles aligned in magnetic field. Magnification 1000x	Lubricant starvation.
 (8). Red Oxides	Red-orange particles aligned in magnetic field. Magnification 450x	Water in the oil or poor lubricant condition.
	Heavy concentration of fine particles at exit of ferrogram™ Magnification 100x	Oil additive depletion.
 8). Cast Iron and Low Alloy Particles	Heat treated ferrogram™ (330°C/625°F) show both straw and blue temper colors indicating medium and low alloy steel respectively. Magnification 400x	Possible camshaft and cylinder wall failure.

TYPICAL MICROSCOPIC WEAR PARTICLES OBSERVED

Actual Photos	Description	Examples of Causes
 (4). Gear Wear	Combined rolling and sliding wear particles. Magnification 200x	Fatigue, scuffing or scoring of gear teeth.
 (5). Bearing Wear	Laminar particle 1µm thick with holes. Magnification 1000x	Rolling contact failure.
 (6). Spheres	Small spheres. <5µm in diameter. Magnification 1000x	Early warning of rolling element bearing failure.

Actual Photos	Description	Examples of Causes
 (10). Aluminum Particle	White metal particle misaligned with magnetic field. Magnification 400x	Aluminum component wear.
 (11). Copper Alloy Particle	Red-yellow particles not aligned with magnetic field. Magnification 400x	Possible beari cage failure.
 (12). Dust/Dirt	Foreign particles of material not characteristic of machine or oil. Magnification 200x	Outside contaminants. Usually sand, dust or dirt.

APPENDIX D. REFERENCES

Required References

1. TB 43-0211, Army Oil Analysis Program, Guide for Leaders and Users, March 1987.

2. TM 38-301-2, Joint Oil Analysis Program Manual, Volume I, 1 September 1987.

3. ASTM-D1744, Annual Book of ASTM Standards, Volume 5.02, Water in Liquid Petroleum Products by Karl Fischer Reagent, 1984.

4. DL 18, Karl Fischer Titrator Operating Instructions.

5. ASTM-D445, Annual Book of ASTM Standards, Vol 5.01, Kinematic Viscosity of Transparent and Opaque Liquids and The Calculation of Dynamic Viscosity, 1984.

6. ASTM-D664, Annual Book of ASTM Standards, Vol 5.01, Neutralization Number by Potentiometric Titration, 1984.

7. MIL-H-6083, Hydraulic Fluid, Petroleum Base, for Preservation and Operation, 1973.

8. MIL-H-83282, Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Aircraft, 1974.

9. MIL-H-46170, Hydraulic Fluid, Rust Inhibited, Fire Resistant Synthetic Hydrocarbon Base, 1982.

10. Operation and Maintenance Instructions Manual for the Fluid Analysis Spectrometer (FAS-2C), MA., September 1985.

11. Ferrograph Operations Manuals, Predict Technologies, Cleveland, Ohio, April 1992.

12. SX FTIR Advanced Operation, Nicolet Analytical Instrument Div., 1990.

TOP 2-2-690
16 August 1996

Forward comments, recommended changes, or any pertinent data which may be of use in improving this publication to Commander, U.S. Army Test and Evaluation Command, ATTN: AMSTE-TM-T, Aberdeen Proving Ground, MD 21005-5055. Phone: (410) 278-1486, DSN: 298-1486 and EMAIL: amstetmt@apg-9.apg.Army.mil. Technical information obtained from the preparing activity: Commander, U.S. Army Aberdeen Test Center, ATTN: STEAC-AC-I, Aberdeen Proving Ground, MD 21005-5059. Phone: (410) 278-4246, DSN: 298-4246, and EMAIL: steacaci@apg-9.apg.Army.mil. Additional copies are available from the Defense Technical Information Center, 8725 John J. Kingman Rd., STE 0944, Fort Belvoir, VA 22060-6218. This document is identified by the accession number (AD No.) printed on the first page.